# LIST OF U.S. CUSTOMS LABORATORY METHODS

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# U.S. CUSTOMS LABORATORY METHODS

## USCL METHOD 20-01

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## AOAC 942.15
## Acidity (Titratable) of Fruit Products

### SAFETY PRECAUTIONS

*This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.*

### 1 SCOPE AND FIELD OF APPLICATION

Chapter 20 of the Harmonized Tariff of the United States (HTSUS) covers preparation of vegetables, fruit, nuts or other parts of plants. This method allows the determination of the total acid content of fruit products.

### 2 REFERENCES

***AOAC 942.15***
Acidity (Titratable) of Fruit Products

# U.S. CUSTOMS LABORATORY METHODS

## USCL METHOD 20-02 INDEX

# Acetic Acid Analysis
# By High-Performance Liquid Chromatography

## SAFETY PRECAUTIONS

*This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.*

0 Introduction

The amount of acetic acid is an important factor for the purpose of heading 2001 of the Harmonized Tariff Schedules of the United States (HTSUS) Chapter 20. This heading includes vegetables, fruits, nuts and other edible parts of plants, prepared or preserved by vinegar or acetic acid. According to the HTSUS, the Customs position as to the minimum amount of acetic acid necessary to determine whether a vegetable is prepared or preserved by vinegar or acetic acid was outlined in Headquarters Ruling Letter (HRL) 069121, dated May 20, 1983 (I/A 247/80). That decision held that a product required a minimum of 0.5 percent acetic acid (subject to allowable tolerances) in the equilibrated product" to be considered as prepared or preserved by vinegar or acetic acid and this position has continued under HTSUS (See HRL Letters, 085838 dated December 21, 1989 and 952738 dated January 27, 1993).
The approach of an acid titration which calculates the total acid as acetic acid, may not always be accurate because many products contain not only acetic acid as preservative, but lactic acid. It should also be taken in consideration that some vegetables can provide certain amount of others acids such as citric acid.

This High Performance Liquid Chromatography (HPLC) method will not only be more accurate than the titration method, but more selective. This method will make it possible to isolate the acetic acid from other acids and identify and/or quantify all the acids present.

### I SCOPE AND FIELD OF APPLICATION

This method applies to vegetables, fruits, nuts and other edible parts of plants, prepared or preserved by vinegar or acetic acid. Common products to be tested are capers, olives, pickles, etc.

### 2 REFERENCES

***AOAC 930.35***
Vinegars

### 3 REAGENTS AND APPARATUS

During the analysis, unless otherwise stated , use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

**3.1** Acetic acid glacial 99.7% ACS Grade, Aldrich Co.

Prepare a 0.5% by weight Acetic Acid Solution. (1.0 ml of Acetic Acid in 200ml of water)

**3.2** Phosphoric Acid 85-87% ACS Grade, Aldrich Co.

**3.3** Acrodisc LC PVDF 0.45Fm filter, Gelman Sciences Co.

**3.4** Chromatograph: Hewlett-Packard High Performance Liquid Chromatogram model 1090

**3.5** Detector : Hewlett-Packard UV/Visible variable Detector model 1050 at 210nm

**3.6** Column: Supelcoil LC-18 Column, 25 cm x 4.6 mm

**3.7** Injector: 30 uL loop

**3.8** Mobile phase is 0.1% Phosphoric Acid

## 4 SAMPLE PREPARATION

**4.1** Perform a physical separation of the liquid contained in the sample. The drained liquid will be used for the analysis.

## 5 PROCEDURE

**5.1** Calibration

**5.1.1** A reference standard should be injected five times using a 0.45Fm filter.

**5.1.2** Reproducibility on the five injections should be verified by calculating the percentage of relative standard deviation (%RSD) for the five individual peak areas. The %RSD should not exceed 2%.

**5.2** Sample Injection

**5.2.1** Samples should be injected in duplicate with two HPLC runs per analytical sample. Use a 0.45Fm filter during the injection.

## 6 EXPRESSION OF RESULTS

**6.1** To calculate the percent weight of acetic acid (by weight):

$$\%\ Acetic\ Acid = \frac{0.5\% * A_S * D}{A_{RS}}$$

Where:

| | | |
|---|---|---|
| $A_{RS}$ | = | the reference standard average area from **5.1** |
| D | = | a dilution factor for the sample (if any) |
| As | = | the area for acetic acid in the sample |

**6.2** To calculate the percent relative standard deviation:

$$\%\ RSD = \frac{standard\ deviation}{average}$$

## 7 BIBLIOGRAPHY

**7.1** Organic Acids Analysis, Supelco Co. HPLC Catalog, page 137.

# U.S. CUSTOMS LABORATORY METHODS

## USCL METHOD 20-03

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# AOAC 945.68
# Canned Vegetables
# Sample Preparation

### SAFETY PRECAUTIONS

*This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.*

### 1 SCOPE AND FIELD OF APPLICATION

Chapter 20 of the Harmonized Tariff of the United States (HTSUS) covers preparation of vegetables, fruit, nuts or other parts of plants. This method gives guidance in the preparation of canned vegetables for analysis.

### 2 REFERENCES

***AOAC 945.68***
Canned Vegetables
Sample Preparation

# U.S. CUSTOMS LABORATORY METHODS

## USCL METHOD 20-04

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# AOAC 968.30
# Canned Vegetables
# Drained Weight

### SAFETY PRECAUTIONS

*This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.*

### 1 SCOPE AND FIELD OF APPLICATION

Chapter 20 of the Harmonized Tariff of the United States (HTSUS) covers preparation of vegetables, fruit, nuts or other parts of plants. This method is used to determine the drained weight of canned vegetables.

### 2 REFERENCES

***AOAC 968.30***
Canned Vegetables
Drained Weight

# U.S. CUSTOMS LABORATORY METHODS

## USCL METHOD 20-05

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# AOAC 932.12
# Solids (Soluble) in Fruits and Fruit Products
# Refractometer Method

### SAFETY PRECAUTIONS

*This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.*

### 1 SCOPE AND FIELD OF APPLICATION

Chapter 20 of the Harmonized Tariff of the United States (HTSUS) covers preparation of vegetables, fruit, nuts or other parts of plants. This method is used to determine the amount of soluble solids in fruits and fruit products.

### 2 REFERENCES

***AOAC 932.12***
Solids (Soluble) in Fruits and Fruit Products
Refractometer Method

# U.S. CUSTOMS LABORATORY METHODS

## USCL METHOD 20-06

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# AOAC 983.17
# Solids (Soluble) in Citrus Fruit Juices
# Refractometer Method

### SAFETY PRECAUTIONS

*This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.*

### 1 SCOPE AND FIELD OF APPLICATION

Chapter 20 of the Harmonized Tariff of the United States (HTSUS) covers preparation of vegetables, fruit, nuts or other parts of plants. This method is used to determine the amount of soluble solids in citrus fruit juices.

### 2 REFERENCES

***AOAC 983.17***
Solids (Soluble) in Citrus Fruit Juices
Refractometer Method

# U.S. CUSTOMS LABORATORY METHODS

## USCL METHOD 20-07

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# AOAC 973.23
# Alcohol in Flavors
# Gas Chromatographic Method

**SAFETY PRECAUTIONS**

*This method does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to its use.*

**1 SCOPE AND FIELD OF APPLICATION**

Chapter 20 of the Harmonized Tariff of the United States (HTSUS) covers preparation of vegetables, fruit, nuts or other parts of plants. This method is used to determine the amount of alcohol in flavors.

**2 REFERENCES**

***AOAC 973.23***
Alcohol in Flavors
Gas Chromatographic
Method